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**Irradiance and Temperature Dependence of Photo-Induced Orientation in Two
Azobenzene-Based Polymers**

by

D. Hore, A. Natansohn and P. Rochon

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**Department of Chemistry
Queen's University
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IRRADIANCE AND TEMPERATURE DEPENDENCE OF PHOTO-INDUCED ORIENTATION IN TWO AZOBENZENE-BASED POLYMERS

Dennis Hore and Almeria Natansohn*

Department of Chemistry, Queen's University, Kingston, Ontario, K7L 3N6

Paul Rochon

Department of Physics, Royal Military College, Kingston, Ontario, K7K 5L0

*author to whom correspondence should be addressed

telephone: (613) 545-2008

facsimile: (613) 545-6669

e-mail: natansoh@chem.queensu.ca

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Dennis Hore and Almeria Natansohn*

Department of Chemistry, Queen's University, Kingston, Ontario, K7L 3N6

Paul Rochon

Department of Physics, Royal Military College, Kingston, Ontario, K7K 5L0

ABSTRACT

This paper concerns itself with photo-induced anisotropy in two azobenzene functionalized polymers. A simple physical model of the process which considers only three possible states of azo chromophores is proposed. This model predicts an equilibrium level of anisotropy which depends on the pump irradiance and temperature. Each of these influences is examined in turn, with experimental illustrations from the two polymers poly[4-[2-(methacryloxy)-ethyl]azobenzene] (pMEA) and poly[4'-[[2-(methacryloxy)ethyl]-ethyl]amino-4-nitroazobenzene] (pDR1M).

keywords: azobenzene polymers, photo-induced orientation, irradiance, temperature

INTRODUCTION

The linking of observable macroscopic properties to molecular-scale mechanisms remains one of the fundamental challenges and enticements of materials science. These properties are often geared toward engineering and technology applications and so are zealously sought. The study of the photo-induced orientation of azobenzene-based polymers represents just such an endeavour.

Anisotropy may be photo-induced in thin films of amorphous polymers containing azobenzene moieties in their side chains. This anisotropy exists well below the glass transition temperature (T_g) in an area as small as a beam can be focussed, exhibits long-range stability, and has high contrast with isotropic regions of the material. These properties make such polymers possible candidates for technologies such as reversible optical storage and waveguides.

Although the details of the orientation mechanism and underlying processes are yet to be agreed upon, a simple physical picture may be proposed. An isotropic distribution of chromophores (as present in an amorphous film) is subject to a linearly polarized pump and individual species are promoted to the lowest energy π^* excited state. One of the relaxation processes from π^* is conversion to the other geometric isomer. Since the *trans* ground state lies lower in energy than that of the *cis* isomer, any *cis* species created revert back to *trans* via a thermal and/or a photochemical route. Therefore in the presence of the pump, many *trans*→*cis*→*trans* isomerization cycles occur, resulting in orientation of the side chains. This motion may continue until the chromophores find themselves in a position where their dipole moments (along the long axis of the molecule) lie perpendicular to the polarization plane of the

pump. Since there can be no further absorption in this orientation, isomerization, and therefore the accompanying side chain motion, stops and the chromophores become "locked". Although steric interactions prohibit the complete orientation of all chromophores, at equilibrium there is a net alignment perpendicular to the pump. This anisotropy manifests itself as a measurable difference in refractive index, or birefringence.

In this study a simple model of photo-induced orientation is presented. The effect of irradiance and temperature on the order parameter is shown for two polymers whose structural units are depicted in Fig. 1. Poly[4'-[[2-(methacryloxy)ethyl]-ethyl]amino-4-nitroazobenzene] (pDR1M) belongs to the pseudo-stilbene spectral class according to Rau's classification scheme (1) since the energy ordering of the (n, π^*) and (π, π^*) states is reversed. For this polymer the bands are practically overlapping, resulting in both *trans*→*cis* and *cis*→*trans* photo-isomerizations at the same excitation wavelength. Poly[4-[2-(methacryloxy)-ethyl]azobenzene] (pMEA) belongs to the azobenzene spectral class (1) with a low intensity (n, π^*) band in the visible and a high intensity (π, π^*) band in the UV. This results in an efficient *cis*→*trans* back-isomerization at 488 nm while the *trans*→*cis* process experiences only the tail of the absorption band.

MODEL

An elementary model of photo-isomerization is based upon the phenomenological mechanism proposed by Sekkat and Dumont (2). There are four dominant processes that contribute to the

orientation. These are: (i) photochemical *trans*→*cis* isomerization, (ii) photochemical *cis*→*trans* back-isomerization, (iii) thermal *cis*→*trans* back-isomerization, and (iv) angular redistribution among species. It is assumed that only the *trans* species may contribute to the observed order parameter on account of its elongated rod-like shape. To make things even simpler, all possible angles at which the side groups can lie are reduced to two: parallel or perpendicular to the pump. As a consequence of the highly delocalized π -electron density of the azo chromophores, the probability that a species will absorb the pump radiation is proportional to $\cos^2\theta$, where θ is the angle between the electronic dipole moment and the polarization vector of the pump. Considering our two orientations, this implies complete absorption for parallel species while perpendicular groups are oblivious to the pump. If the back-isomerization is allowed to proceed by the thermal route only, an identical solution for the *photostationary* order parameter is obtained with and without the photochemical back-isomerization. Therefore, for the primary purpose of representing this equilibrium, only the thermal back-isomerization will be included in the model, keeping in mind that both occur in the actual experimental system. With these considerations in mind three populations have to be monitored: *cis* (C), *trans* parallel to the pump (T_{\parallel}), and *trans* perpendicular to the pump (T_{\perp}). The pump I^* creates *cis* species which thermally relax back to *trans* with a reciprocal lifetime of γ . *Trans* species may flip back and forth between parallel and perpendicular orientations with a diffusion constant Γ .

The model is implemented by means of three coupled differential equations which govern the change in each population with time.

$$[1] \quad \frac{dT_{\parallel}}{dt} = -I^*T_{\parallel} + \frac{1}{2}\gamma C - \Gamma T_{\parallel} + \Gamma T_{\perp}$$

In the first equation, the T_{\parallel} population is being diminished by the pump and by rotation to the perpendicular direction; increased by *cis*→*trans* thermal relaxation and rotational diffusion from the perpendicular towards the parallel position. The thermal *cis*→*trans* process is here assumed to equally populate T_{\parallel} and T_{\perp} .

$$[2] \quad \frac{dT_{\perp}}{dt} = +\frac{1}{2}\gamma C + \Gamma T_{\parallel} - \Gamma T_{\perp}$$

In the second equation, the number of T_{\perp} chromophores is increased by thermal back-isomerization and is also susceptible to rotational diffusion.

$$[3] \quad \frac{dC}{dt} = +I^* T_{\parallel} - \gamma C$$

In the last equation, the *cis* species is increased in number by pumping from *trans* and suffers losses to the thermal back-isomerization. An energy level diagram for such a system is shown in Fig. 2.

EXPERIMENTAL

The synthesis of pMEA (3) and pDR1M (4) has been described in previous publications. The polymers were spin-cast from THF onto glass substrates and then held at their glass transition temperatures (120°C for pDR1M and 80°C for pMEA) for one hour. This served to adhere the matrix onto the substrate, drive out solvent, and remove any anisotropy which may have been induced by spin-coating. Film thickness was measured using a Sloan Dektak II profilometer. Before beginning any of the experiments, it was necessary to choose films of an appropriate maturity to obtain reproducible rates and saturation levels, and there has been a similar report of such an age requirement in the literature (5). Consistent results were achieved with films left 2 - 4 weeks at room temperature following preparation.

A schematic of the optical bench is given in Fig. 3. In the detection circuit a diode laser operating near 690 nm was incident onto a linear polarizer at 45°. The beam then passed through a focussing lens, the sample, and a crossed polarizer before reaching the detector, a photomultiplier tube. The square root of the signal from the detector is proportional to the phase lag suffered by the probe through a birefringent sample. Refractive index difference ($\Delta n = n_{\parallel} - n_{\perp}$) was then calculated using

$$[4] \quad \Delta n = \frac{\sqrt{I} \Delta \varphi}{\sqrt{I_{ref}} k d}$$

where I is the signal from the amplifier, $\Delta\phi$ is the phase lag at reference, d is the sample thickness, $k = 2\pi/\lambda$ is the wavevector, and I_{ref} is the reference signal used in the calibration.

From this, the order parameter S was obtained using

$$[5] \quad \langle S \rangle = \frac{\Delta n}{3n_{band}}$$

where n_{band} refers to the effective isotropic index of refraction of the chromophores alone. Since the birefringence is likely the result of an anisotropic distribution of azo side groups, a measure of their orientation should include the contribution to the refractive index of just those groups. This was obtained from the (π, π^*) and (n, π^*) absorption bands using a Kramers-Kronig relation

$$[6] \quad n_{band}(\lambda) = \int_{band} \frac{\alpha(\lambda')}{1 - (\lambda'/\lambda)^2} d\lambda'$$

where $\alpha(\lambda')$ is the absorption coefficient. For pDR1M this gave a photostationary order parameter of -0.13 , in agreement with that obtained by dichroic infrared (6) and transient absorption spectroscopy (7).

The 488 nm line of an Ar^+ laser was used as an excitation source, intersecting the probe beam at the sample surface. A Pockels cell was used to vary the laser polarization between linear and circular. A typical experimental protocol involved irradiating the sample for 200 s while collecting data at a rate of 1 point/s for the first 10 s, followed by 10 points/s for the remaining

time. An Instec temperature controller and heating stage were used to maintain temperatures accurate to within $\pm 0.1^\circ\text{C}$.

RESULTS AND DISCUSSION

Model Predictions

When a simulation of photo-orientation based on the above model is run, the macroscopic order parameter S reaches a saturation value, as is observed experimentally (Fig. 4). It is possible to derive an analytical expression for this saturation value by solving the linear system (eqs. [1] - [3]) with the steady-state approximation, giving

$$[7] \quad \langle S \rangle = \frac{T_{\parallel} - T_{\perp}}{T_{\parallel} + 2T_{\perp}} = -\frac{I^*}{6\Gamma + 2I^*}.$$

Although the model assumes the orientation to be two-dimensional (in the plane of the film), the order parameter must approximate a three-dimensional system to be compared with the experimental value given by eq. [5]. For the calculation of S , the total *trans* population occupies 3 states. Here an additional T_{\perp} exists (chromophores oriented normal to the film surface and hence also perpendicular to the pump) which is for simplicity assumed to have the same population as the original in-plane T_{\perp} . The result is nearly identical to that obtained by

Labarthe and Sourisseau (7) in their implementation of Sekkat and Dumont's model (2) obtained with a more complete theory where all angular distributions are taken into account.

The model predicts a photostationary order parameter that is independent of the *cis* lifetime γ . The only influences which remain are the pumping rate I^* and the *trans* diffusion constant Γ . It is possible to obtain an estimate of the pumping rate based on the *trans*→*cis* quantum efficiency Φ and experimentally measurable *trans* absorption cross-section σ .

$$[8] \quad I^* = I_{\text{pump}} \Phi \sigma$$

The likelihood of photon absorption leading to isomerization is directly related to the free volume available for such a configurational change, and so the quantum efficiency is sensitive to the polymer matrix. It has been shown to vary by more than four orders of magnitude from solution values of 0.25 (1) to 0.11 for doped systems (8) to 7.4×10^{-6} in Langmuir-Blodgett films (9). Our modelling suggests a quantum efficiency in the range 5×10^{-3} to 5×10^{-2} which is low but reasonable, considering the inevitable crowding in a fully chromophore-loaded functionalized system. The absorption cross-section was estimated based on the absorbance, film thickness, and chromophore density. Such a calculation gives a value of $\sigma = 1.3 \times 10^{-18} \text{ cm}^2$ for pMEA and $\sigma = 1.2 \times 10^{-16} \text{ cm}^2$ for pDR1M. The other parameter of interest here, the *trans* diffusion constant Γ , will be discussed along with the temperature results.

Effect of Pump Irradiance

It is of primary interest to compare the experimental results of the effect of pump irradiance on the photostationary order parameter with those predicted by eq. [7]. For pump irradiances in the range 13 mW/cm^2 to 60 mW/cm^2 , the order parameter remained constant at -0.13 for pDR1M and increased in absolute value for pMEA (Fig. 5) in qualitative agreement with eq. [7]. This variation for pMEA, however, was slight (less than 5%) so the dependence is weak. Still, the adherence of pMEA and disagreement of pDR1M with the predicted irradiance behaviour suggest that dipolar interactions in pDR1M may be the cause of the observed differences. This idea will be discussed further in the following section. Below 10 mW/cm^2 , the absolute saturation level decreases with irradiance for both polymers. Unfortunately, the long time required to reach equilibrium at such low pump irradiances (ca. 4 hours at 5 mW/cm^2) makes a quantitative investigation of the relationship impractical by these methods. A similar study on the same chromophore as contained in pDR1M (Disperse Red 1) doped into a poly(methyl methacrylate) matrix revealed changes in the attainable orientation when pumping below 100 mW/cm^2 (7). This larger saturation order parameter is to be expected for a doped system due to a much lower chromophore density.

The normalized order parameter (S_n) vs time curves at each irradiance were fitted to the biexponential equation

$$[9] \quad S_n = A [1 - \exp(-k_a t)] + B [1 - \exp(-k_b t)]$$

where A and B represent the amplitudes of the two exponentials, k_a and k_b the rate constants. The fitting indicates that for both polymers k_a and k_b increase with increasing irradiance. This is shown for pMEA in Fig. 6 and for pDR1M in Fig. 7. The amplitude A of the term describing the fast process increases as well, slightly faster for pMEA than for pDR1M at lower irradiances (Fig. 8). Song *et al.* carried out similar studies on an azobenzene-functionalized polymer (5) over the same range of pump irradiances. Although there are slight differences in the fitting procedure, their results showed no change in rate constant for different pump irradiances. Their data also shows that the amplitude of the exponential describing the fast process increases linearly with pump irradiance. The three values of irradiance used in that study are indicated with arrows in Fig. 8, illustrating that this region may appear to be linear for our samples as well. Measurements at lower irradiances, however, reveal that the contribution of the fast process decreases sharply in favour of the slower one.

Effect of Temperature

For both pMEA and pDR1M, the photostationary order parameter increases monotonically as a function of temperature, approaching zero as the systems restore their isotropic distribution of chromophores around T_g (Fig. 9). Here the influence and temperature dependence of the *trans* rotational diffusion constant Γ may be seen. At higher temperatures there is more redistribution and a greater tendency towards isotropy. When Γ becomes very large, eq. [7] predicts that the order parameter S tends towards zero. Examining the other limit, as $\Gamma \rightarrow 0$, $S \rightarrow -0.5$, the maximum order achievable in theory. That this may never be fulfilled is largely the result of steric factors. The magnitude of Γ is, however, not as closely related to the absolute temperature

as it is to the glass transition temperature, for it is above T_g that polymers find the maximum free volume available to redistribute their pendant chromophores. This suggests a comparison on the basis of a reduced temperature, such as $T - T_g$ (Fig. 10). Although the manner in which both polymers lose their ability to maintain ordered chromophores appears markedly different in Fig. 9 (linear in the case of pMEA, sigmoidal for pDR1M), Fig. 10 shows that for a temperature region in which both systems may be compared (55° below to 5° above their respective T_g) the behaviour is remarkably similar.

A surprising result came from fitting the normalized order parameter curves to the biexponential described by eq. [9]. For both polymers, the variation in rate constant (k_b) with temperature showed no trend for the slow process, the data being completely scattered. The rate constant (k_a) for the fast process, however, increased with increasing temperature for pMEA and decreased for pDR1M (Fig. 11). This behaviour is in contrast to the rate constant dependence on irradiance discussed in the previous section where rates for both polymers increase with increasing pump irradiance. A possible explanation might involve the larger dipole moment of the donor-acceptor substituted pDR1M (10). Large dipole-dipole interactions have been shown to give rise to cooperative effects (11) where neighbouring groups participate in orientation in an effort to minimize the electronic repulsion in energetically unfavourable chromophore alignment.

These results are best considered with the two competing processes involved in mind. On the one hand there is alignment due to the polarization of the laser; on the other there is a tendency towards disorder and restoration of the equilibrium isotropy. Larger pump irradiances would favour the orientation process and have very little effect on thermal randomization, as was demonstrated experimentally. Increasing the temperature, however, would increase the rates of

both processes due to an enhanced mobility accompanying an increasing free volume. The interplay between the two processes at higher temperatures is likely sensitive to dipolar interactions, resulting in a rate constant which decreases on heating for pDR1M. Since such electronic interactions are much weaker in pMEA, the resulting behaviour is different. A separate study to verify this hypothesis is in progress analyzing copolymers and blends with different orientations of the dipoles.

SUMMARY AND CONCLUSIONS

The photo-induced reorientation of azobenzene-based polymers is a complex phenomenon, especially in the case of functionalized polymers, since there are many possible interactions between the chromophores and the matrix. The simple model presented here accounts for experimentally observed order parameters using optical pumping and thermal redistribution. It has been used to arrive at an expression for the photostationary order parameter in terms of the pumping rate and the *trans* rotational diffusion constant. The predicted irradiance dependence of the orientation process has been qualitatively verified for pMEA, while pDR1M appears to be less dependent on the pump intensity. The rotational diffusion constant is a function of temperature and causes the photostationary order parameter to decrease at higher temperatures. For the polymer pDR1M, strong dipole-dipole interactions may be responsible for an orientation rate constant that decreases at higher temperatures. All of the experimental evidence suggests that the model of photo-induced orientation is only sufficient to describe a system such as pMEA.

where the lack of strong dipolar interactions allows the chromophores to be aligned independently, similar to the situation in a doped system with low chromophore density. For a system such as pDR1M, electronic effects such as dipole-dipole interactions will have to be taken into account.

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Macromolecules, in press

FIGURE CAPTIONS

Fig. 1. The structural units of pMEA (top) and pDR1M (bottom).

Fig. 2. An energy level diagram for the 3-state model of photo-induced reorientation. Here promotion to π^* is shown only for the photochemical *trans*→*cis* isomerization. *Cis* species thermally relax to *trans*, and redistribution among *trans* species is also allowed to occur.

Fig. 3. A schematic of the optical bench used to induce and measure birefringence.

Fig. 4. The photostationary order parameter attained by an experimental system (pDR1M) and by simulation with the model.

Fig. 5. The variation in photostationary order parameter with pump irradiance for pMEA.

Fig. 6. (a) Fast process rate constant k_a vs irradiance and (b) slow process rate constant k_b vs irradiance for pMEA.

Fig. 7. (a) Fast process rate constant k_a vs irradiance and (b) slow process rate constant k_b vs irradiance for pDR1M.

Fig. 8. Amplitude of the fast process vs irradiance for pMEA and pDR1M.

Fig. 9. The variation in photostationary order parameter for pMEA and pDR1M with increasing temperature.

Fig. 10. Photostationary order parameter plotted against the difference between experimental temperature and sample T_g , shown for a range over which both polymers may be compared.

Fig. 11. The temperature behaviour of the rate constant describing the fast process (k_a) for pMEA and pDR1M.

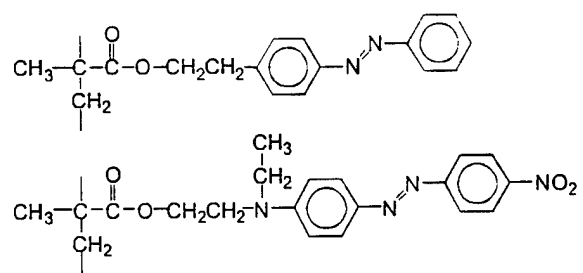


Fig 1

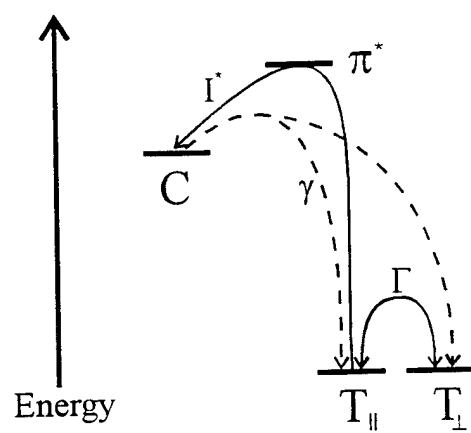


Fig. 2

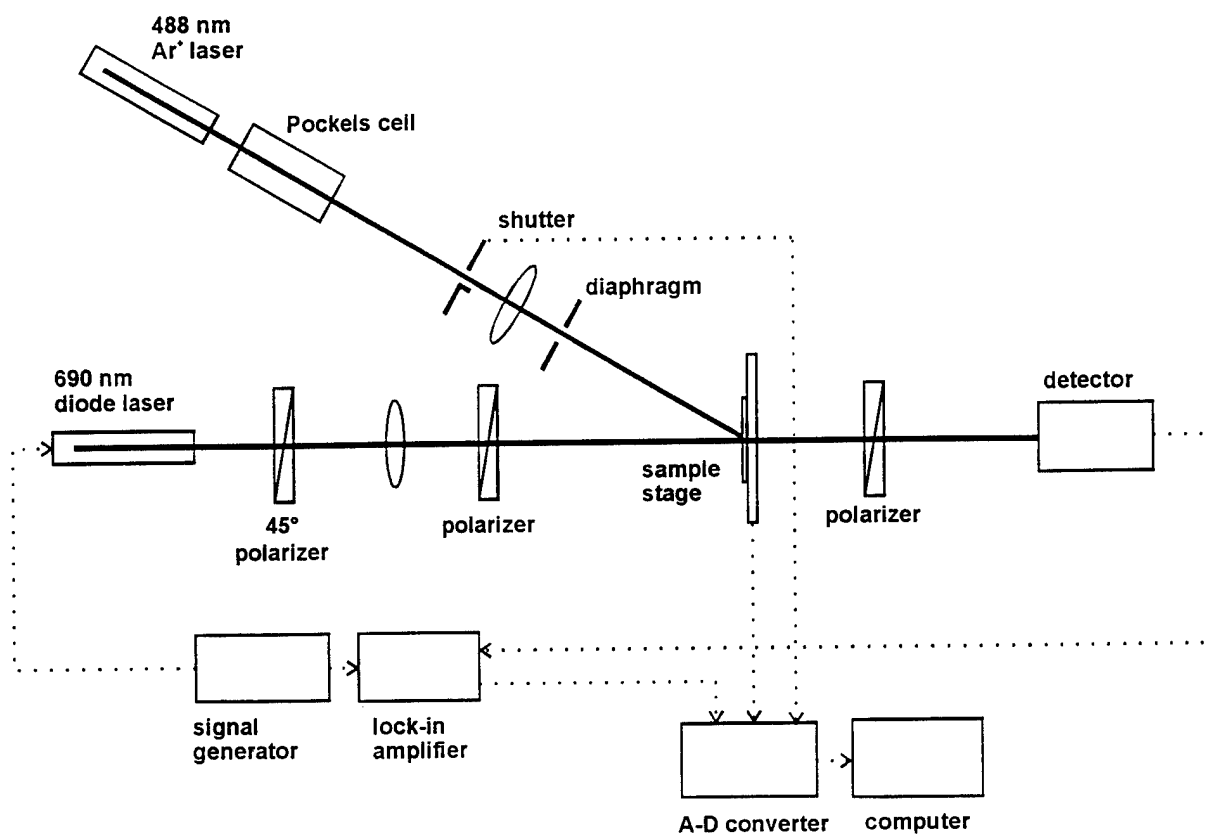


Fig. 3

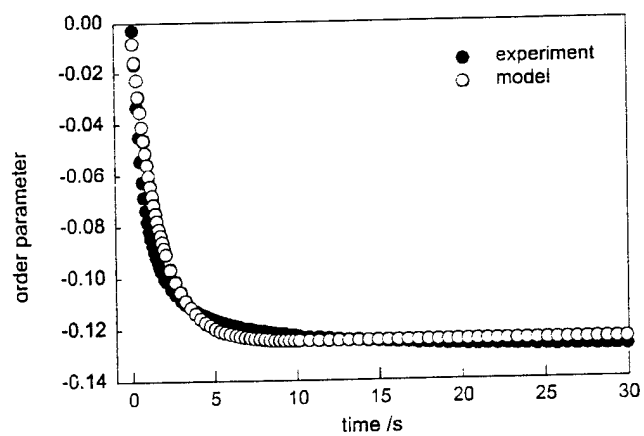


Fig. 4

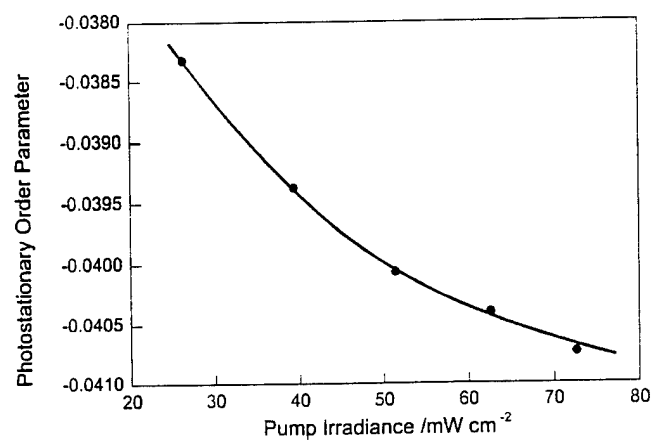


Fig. 5

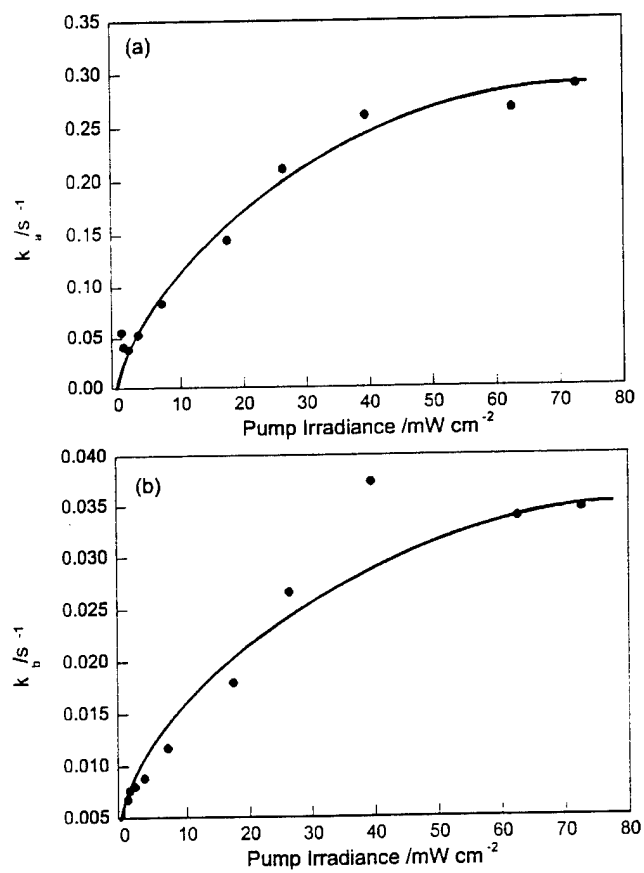


Fig. 6

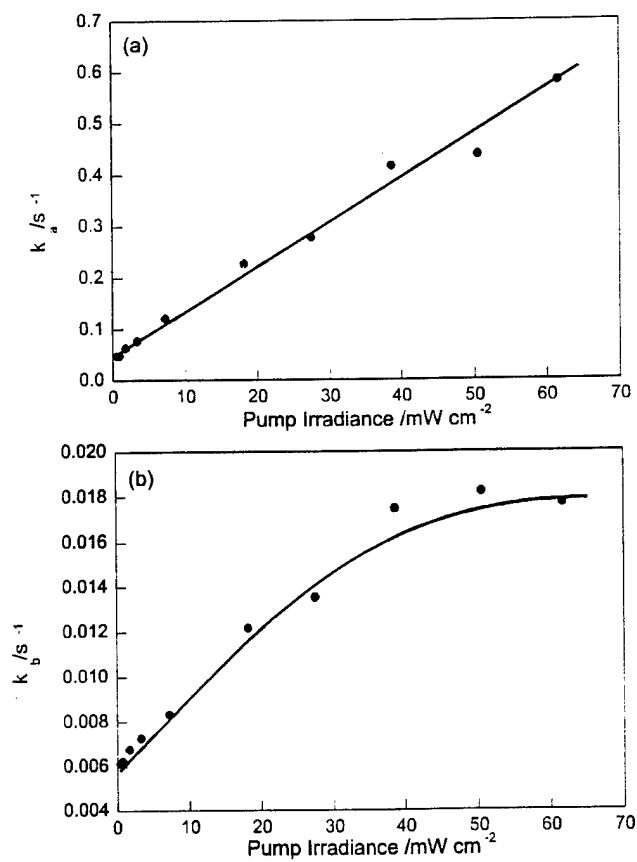


Fig. 7

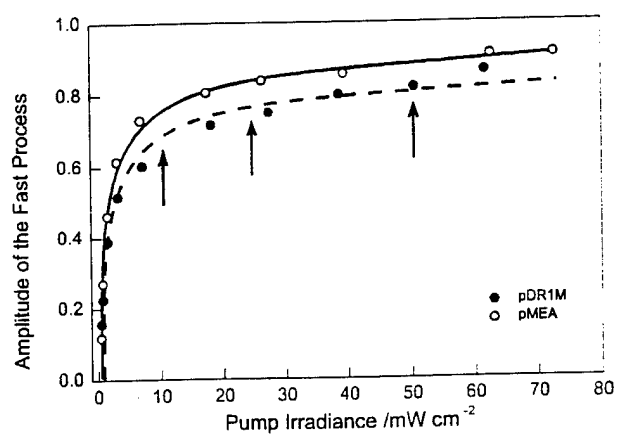


Fig. 8

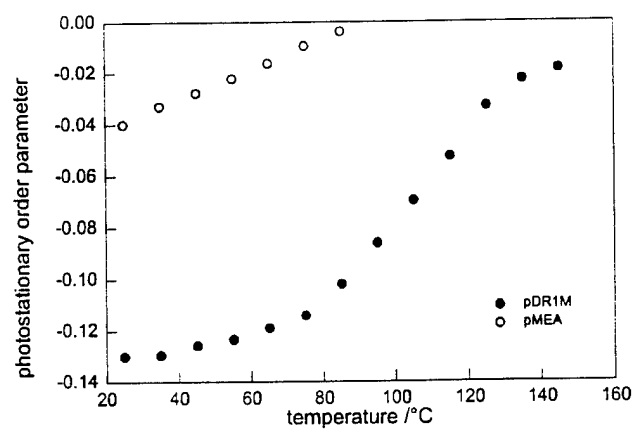


Fig. 9

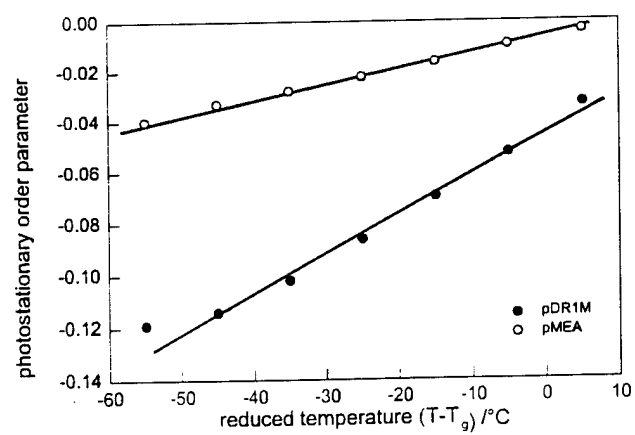


Fig 10

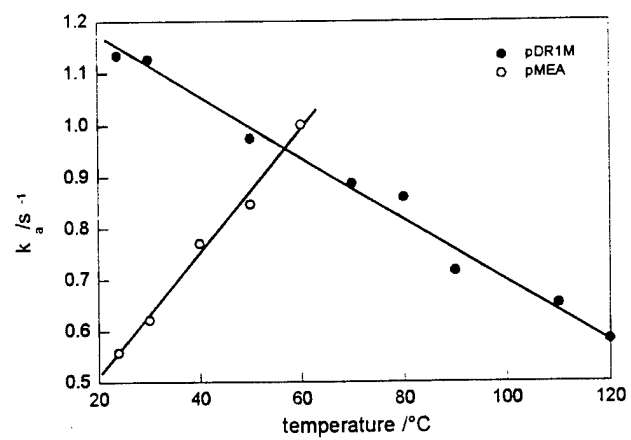


Fig. 11

IRRADIANCE AND TEMPERATURE DEPENDENCE OF PHOTO-INDUCED ORIENTATION IN TWO AZOBENZENE-BASED POLYMERS

Dennis Hore and Almeria Natansohn

Department of Chemistry, Queen's University, Kingston, Ontario, K7L 3N6

Paul Rochon

Department of Physics, Royal Military College, Kingston, Ontario, K7K 5L0

